PROJECT ADMINISTRATION DATA SHEET

[X] ORIGINAL [ ] REVISION NO. _____

Project No. E-26-602 GTRI/~~GTX~~ DATE 3 / 13/84

Project Director: Dr. G. G. Eichholz School/~~Lab~~ NE

Sponsor: E. I. duPont de Nemours & Co., Aiken, S.C.

Type Agreement: P.O. AX-0630772 (DOE Prime DE-AC09-76SR00001)

Award Period: From 1/2/84 To 12/31/84 (Performance) 12/31/84 (Reports)

Sponsor Amount:	This Change	Total to Date
Estimated:	$ 49,782	$ 49,782
Funded:	$ 49,782	$ 49,782

Cost Sharing Amount: $ None Cost Sharing No: N/A

Title: Environmental Impact of Buried Metallic Mercury

ADMINISTRATIVE DATA OCA Contact William F. Brown x-4820

1) Sponsor Technical Contact:

Dr. John A. Stone
E. I. du Pont de Nemours & Co.
Savannah River Plant
Aiken, S.C. 29808-0001
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2) Sponsor Admin/Contractual Matters:

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Aiken, S.C. 29808-0001
(803) 725-1008

Defense Priority Rating: N/A Military Security Classification: N/A

(or) Company/Industrial Proprietary: N/A

RESTRICTIONS

See Attached _____ Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval — Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with None proposed or anticipated.

COMMENTS:

Sponsor ID #02.010.128.84

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FORM OCA 4:383

SRH28 296

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date 1/28/85

Project No. E-26-602 School/~~Lab~~ NE

Includes Subproject No.(s) N/A

Project Director(s) Dr. G. G. Eichholz GTRI / ~~GIT~~

Sponsor E. I. duPont de Nemours & Co., Aiken, S. C.

Title Environmental Impact of Burial Metallic Mercury

Effective Completion Date: 12/31/84 (Performance) 12/31/84 (Reports)

Grant/Contract Closeout Actions Remaining:

- [ ] None
- [XX] Final Invoice or Final Fiscal Report
- [ ] Closing Documents
- [XX] Final Report of Inventions
- [XX] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other

Continues Project No. Continued by Project No.

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Form OCA 50:1028

E-26-602

Georgia Institute of Technology
A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA
SCHOOL OF NUCLEAR ENGINEERING AND HEALTH PHYSICS
ATLANTA, GEORGIA 30332
(404) 894-3720

February 8, 1984

Dr. John C. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. DuPont deNemours & Company
Aiken, SC 29808

First Monthly Progress Report - Project E26-602

Dear Dr. Stone:

Following our initial discussions last month with Dr. Oblath and yourself at SRL, work has concentrated on a literature survey and a review of current activities elsewhere. Mr. Robert Kury has been taken on as a graduate research assistant to help with this work. Discussions with Dr. Ralph R. Turner at Oak Ridge National Laboratory, and others there, reinforce our belief that the principal transport mechanism of concern is by the adsorption of mercury on suspended colloids that may then move through subsurface aquifers.

At this time we are planning experimental work on gravitational diffusion of metallic mercury through "re-presentative" soil columns as well as adsorption measurements of mercury on kaolin suspensions. We have considerable past experience in the area of particulate transport through subsurface waters which should prove relevant here.

Our literature survey is progressing well. We are beginning the process of comparing the Orebaugh and Hale report with the current knowledge base.

Please call me if you have any questions or comments.

Sincerely,

Geoffrey G. Eichholz
Regents' Professor

cc: Dr. J.L. Carden
Mr. W.F. Brown (OCA)

E-26-602

Georgia Institute of Technology
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(404) 894-3720

March 7, 1984

Dr. John C. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. DuPont deNemours & Company
Aiken, SC 29808

Second Monthly Progress Report - Project E26-602

Dear Dr. Stone:

Our literature search has continued to be fruitful. We have now reviewed the literature supporting the Orebaugh and Hale report as well as a number of articles published since its submission. The most interesting work post-dating Ocbaugh and Hale deals with mercury transport on soil colloids. We are also looking for studies dealing with the recovery of mercury by the cold vapor technique when soil colloids are present in solution. We plan to complete our search by the end of this week and begin preparing a report on our findings for submission by the end of this month.

Estimates on movement of mercury through porous media, under gravity only, have been received from Dr. R.R. Turner at Oak Ridge National Laboratory, who also drew our attention to some relevant reports.

The principal experimental effort over the coming weeks will be directed to measurements of mercury adsorption on suspended clay particles and the stability of the sorbed mercury.

Yours sincerely,

G.G. Eichholz
Regents' Professor

cc: J.L. Carden
W.F. Brown (OCA)

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Third Monthly Progress Report, April 1984

E-26-602

Georgia Institute of Technology

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May 9, 1984

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, SC 29808

Fourth Monthly Progress Report - Project E26-602

Dear Dr. Stone:

The work for the past month has concentrated on the completion of the critical review of the literature on mercury migration in the environment and its application to conditions at the Savannah River Plant. This report in the form of a topical progress report, is attached herewith and confirms our preliminary conclusions that major migration of mercury from the disposal area into the water table is fairly improbable. It does indicate several areas, where additional work would seem desirable, such as improvements in analytical procedures, experimental tests on uptake and mobility on colloids and other particulates, and sorption/desorption interaction with SRP soil.

Several different soil columns have been set up to demonstrate the degree of mercury seepage as a function of pore size and soil type. They will be subject to observation over an extended period. We are also trying to design an effective test to measure mercury attachment on clay particles suspended in water.

We would welcome your comments on the bibliography and our further directions into experimental work.

Yours sincerely,

Geoffrey G. Eichholz,
Regents' Professor

GGE/swm

cc: J.L. Carden
O.H. Rodgers (OCA)

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June 11, 1984

Please reply to:

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E. I. Du Pont de Nemours & Company
Aiken, SC 29808

Fifth Monthly Progress Report - Project E26-602

Dear Dr. Stone:

During the past month we have completed and dispatched to you the literature review, as a Project Topical Report, which Dr. Carden discussed with you when he visited SRP last week. We are planning to keep up with the literature, though in a less intensive fashion.

I look forward to discussing with you the more experimental phase of this project later on this month. As Dr. Carden told you we have categorized this work into the following aspects:

A. Solubility Studies
Solubility (thermodynamic) studies on mercury in water, both pure and soil-equilibrated, and in the presence of various chelating agents.

B. Percolation tests
Movement of mercury through soil columns and absorption measurements on soil and minerals

C. Colloid Uptake
Absorption measurements on iron oxide colloids, kaolinite and SRP clays in direct contact and in aqueous suspensions.

D. Atomic Absorption Analysis
Design and Evaluation of a cold vapor apparatus. Calibration and development of procedures.

E. Neutron Activation Analysis
Comparison with AA results and application to colloidal sorption samples.

Work has already been initiated in some of these areas, notably item B, and construction of the cold vapor cell will be started shortly. Some of these tests we can discuss in more detail when I see you on June 27.

Yours sincerely

G.G. Eichholz
Regents' Professor

GGE/swm

cc: J. L. Carden
O. H. Rodgers (OCA)

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July 10, 1984

Please reply to:

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, SC 29808

Sixth Monthly Progress Report - Project E-26-602/E-25-626

Dear Dr. Stone:

The principal event of the past month were two visits to SRL to discuss the project, one by Dr. Carden on June 7, and one by myself on June 27, 1984. These meetings helped to clarify the immediate course of the project. In particular, it added a task to measure the absorption of metallic mercury on structural materials and we are just getting under way to plan that work.

The cold-vapor apparatus has been designed and is currently being built to easily adapt to an atomic absorption spectrophotometer. The apparatus will be tested and modifications made to optimize conditions which will be capable of a sensitivity in the parts per billion range. The system will then be calibrated with prepared solutions and Fischer Standards to verify sensitivity and accuracy. The apparatus will serve as a detector system for the mercury colloid sample being prepared.

We are in the process of preparing some radioactive mercury (Hg-203) for tracer tests on soil percolation, surface absorption and solubility measurements.

Yours sincerely,

G.G. Eichholz
Regents' Professor

cc: J.L. Carden
O.H. Rodgers (OCA)

GGE/sm

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August 10, 1984

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, SC 29808

Seventh Monthly Progress Report - Project E-26-602/E-25-626

Dear Dr. Stone:

During the past month the cold-vapor cell for the atomic absorption analysis system has been built and the associated components have been assembled. The system is undergoing tests and we expect to calibrate it in the next two weeks.

Preliminary test have begun on the adsorption of mercury on coupons of various structural materials and we expect to select a reproducible procedure shortly. The mercury we are using may not be clean enough and we are taking steps to obtain commercially purified metal. Test work on the dispersion, suspension or dissolution of mercury in groundwater and organic contaminants are getting under way rather slowly. We expect to activate some mercury in the Georgia Tech reactor next week for use as a radioactive tracer.

Ms. Kim Smith and Mr. Steven Musolino have joined the project for the summer.

Yours sincerely,

G.C. Eichholz
Regents' Professor

GGE/swm

cc: J. L. Carden
O. H. Rodgers (OCA)

E-26-602

Georgia Institute of Technology

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September 7, 1984

Please reply to:

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, SC 29808

Eighth Monthly Progress Report - Project E-26-602/E-25-626

Dear Dr. Stone:

Progress on the project has been relatively slow during the month. The atomic absorption system has been run a few times but at this stage its sensitivity for mercury is not as good as expected and we are trying to find the cause.

Three mercury samples were prepared and flame-sealed in quartz tubes for activation. A sample was activated and assayed for radioactive impurities. No identifiable peaks other than those of the mercury isotopes were found. The active sample is kept ready for tracer tests.

Several tests were started to measure Hg absorption on coupons of laboratory materials and these are under way. Some difficulties are anticipated in removing the samples from the test solution for counting and we are exploring alternative procedures. We have also assembled materials for Hg solubility tests and expect to start these later this month.

Mr. S. Musolino and Miss Kim Smith have left the project at the end of the summer quarter and we expect to start two new research assistants when the fall quarter starts.

Yours truly

Geoffrey G. Eichholz

cc: J.L. Carden
O.H. Rodgers (OCA)

E-26-602

Georgia Institute of Technology

A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA

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October 9, 1984

Please reply to:

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ATLANTA, GEORGIA 30332 U.S.A.

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, SC 29808

Ninth Monthly Progress Report - Project E-26-602/E-25-626

Dear Dr. Stone:

Additional work has been done during the month to improve the sensitivity of the atomic absorption analysis system. Improvements were obtained by changing the method of sample injection; however, a minor error still arises from the presence of small air bubbles in the syringe tip and attempts are being made to minimize this.

The coupon absorption tests have been moved to a different location, now in the Emerson Building, and we are starting a new series of tests. The previous tests showed appreciable film formation on galvanized iron, less so on other materials. The main problem in long - term tests arises from the formation of surface scum on the liquid samples and we are exploring the use of a liquid cover to minimize this.

A new series of solubility tests and emulsion formation studies is also getting under way and we are introducing the new group members to the work. Matthew McFee and Jonathan Newman have joined the program.

We will discuss progress in this work when you and Steve Oblath visit here on October 30. Please call me if you have any questions.

Yours sincerely,

G.G. Eichholz
Regents' Professor

cc: Dr. Carden
O.H. Rodgers (OCA)

E-26-602

Georgia Institute of Technology

A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA

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November 5, 1984

Please reply to:

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E. I. DuPont de Nemours & Company
Aiken, SC 29808

Tenth Monthly Progress Report - Project E-26-602/E-25-626

Dear Dr. Stone:

Work progressed steadily during the past months in the three areas of current concern, atomic absorption analysis, fluid uptake of liquid mercury and surface interaction with coupons of structural materials. Since the present status was reviewed with you during your visit here last week, there appears to be no reason to go into further details.

We have drawn up a proposed program for the coming year to cover continued work on this project under a new contract. A draft project proposal is enclosed herewith for your review and we hope that it agrees fairly well with the scope envisaged by you. We shall look forward to your formal RFQ at an early date.

In the meantime we are starting to prepare the Final Report for the current project to be completed by mid-December.

Please call me if you have any questions regarding the current work or the draft proposal.

Yours sincerely

G. G. Eichholz
Regents' Professor,

GGE/swm

cc: J.L. Carden
O.H. Rodgers (OCA)
J. Brighton

Telephone: 404-894-3720 Telex: 542507 GTRIOCAATL Fax: 404-894-3120 (Verify: 404-894-4850)

ENVIRONMENTAL IMPACT OF BURIED METALLIC MERCURY

FINAL REPORT

26-602

PROJECT E25-626

SRP Purchase Order AX-0630772

edited by

Geoffrey G. Eichholz

Project Director

Submitted to

Waste Disposal Technology Division

Savannah River Laboratory

EI DuPont de Nemours & Co.

Aiken, S.C. 29808

December 1984

CONTENTS

SUMMARY

The work described in this report was undertaken in support of current studies conducted by the Savannah River Laboratory on any risks associated with the presence of some metallic mercury known to have been buried with other wastes in certain on-site disposal trenches. The purposes of this investigation was to establish current knowledge in this field, to study possible migration mechanisms, and to arrive at a reasonable impact assessment on possible groundwater contamination by this material.

The work described here, which is being continued, has addressed four related, but distinct tasks:

1. A literature survey on mercury entrainment, migration and analysis, which was completed and submitted in May 1984 as a Topical Report.
2. Experimental tests on the mobility and uptake in fluids of massive and disperse elemental mercury.
3. Development of analytical facilities for the determination of trace quantities of mercury in environmental samples.
4. A study of the behavior of structural materials in the presence of trace amounts of mercury.

Since most of this work is continuing, this report functions essentially as a progress report.

PROJECT PERSONNEL

(all part-time)

Geoffrey G. Eichholz, Ph.D.	Project Director
John L. Carden, Ph.D.	Adjunct Assoc. Professor
Robert F. Kury, B.S.	Graduate Research Asst.
Dean L. Gross, B.S.	Student Assistant
Stephen V. Musolino M.S.H.P.	Graduate Student Asst.
Deborah K. Smith, B.S.	Graduate Student Asst.
Matthew C. McFee, B.S.H.P.	Graduate Student Asst.
Jonathan R. Newman, B.S.	Graduate Student Asst.

INTRODUCTION

Mercury has long been recognized as a toxic industrial material and a great deal has been written about the hazards from mercury spills and mercury vapor in industrial operations. Its effects in environmental settings were less clearly recognized, since elemental mercury was long considered relative immobile in soil and not very soluble in water. This situation changed in 1968 - 1970 when substantial concentrations were found in fish, first in Sweden and later in the Great Lakes. Contaminating sources were primarily chloralkali plants which dumped large quantities of metallic mercury daily, as well as some pulp and paper plants (1). In its worst form of mercury poisoning, as Minamata disease, mercury wastes were identified as the cause of extensive congenital cases of mental defects and other problems. Many other cases of mercury poisoning were due to organic mercury, often dispersed in fungicides, and the toxic effects from contaminated fish have been ascribed to bacterial conversion of metallic mercury to methyl mercury, which is much more soluble and readily assimilated in the body (2). In the absence of oxygen, inorganic mercury is converted to the toxic methyl mercury $(CH_3)_2$ Hg) by the bacterium methanobacterium Amelanskis.

A great deal of work was done to analyze fish, in particular, to identify the sources of mercury. The general concern diminished somewhat,

when it was found that mercury is actually widely distributed in nature (1). In soil it is present at levels of 0.05 ppm, in coal 0.09-33 ppm and about 3000 tons are released annually throughout the world, mainly from natural cinnabar (HgS) in volcanic rocks. Millions of mercury thermometers are broken annually in Canada alone (3) and the occupational "mad hatters disease" in the felt industry is well-known (4).

Concern about environmental mercury was rekindled when Science reported in the fall of 1982 that substantial mercury contamination had been found at Oak Ridge, Tennessee. The Department of Energy initiated a major project to localize and analyze any mercury occurrences there and identify their sources. Most of the contamination there has been ascribed to soluble waste mercury disposed of in effluents from the Y-12 Plant, which reached the Clinch River. River silt and dredgings were then used for landfill and contaminated soil and groundwater were identified in many locations(5).

At the Savannah River Plant it was known that some metallic mercury was buried in several locations and Orebaugh and Hale, in 1976, conducted an extensive study on mercury transport. They considered the effects of gaseous diffusion of mercury vapor to the atmosphere and concluded that colloidal suspension is the dominant mode for transport of mercury from the burial site. They estimated that mercury from this source even under worst-case conditions would contribute only amounts in the range of natural concentrations to local surface streams.

The present project had several objectives:

1. To update the knowledge on migration and to compile a critical review of the literature;
2. To study solubility and absorption effects of elemental mercury in low concentrations in water and waste fluids;
3. To assist in the selection of structural materials for possible application in lysimeter studies on mercury migration.

The first task was completed at an early stage in the project; the other two are continuing beyond the termination of the current project.

In support of the above tasks, considerable effort was devoted to the development of a cold-vapor cell for low-level analysis of environmental mercury. Work was also done to prepare radioactive Hg-197/203 for tracer tests.

LITERATURE REVIEW

The first task of this project was the compilation of an extensive literature review on mercury migration. This review was submitted as a Topical Report to the Savannah River Laboratory in May 1984 (7). The material was organized into eight categories:

1. Chemical states of mercury under environmental conditions;
2. Diffusion of mercury vapor through soil;
3. Solubility and stability of mercury in environmental waters;
4. Transport of mercury on colloids;
5. Models of mercury migration through the environment;
6. Analytical techniques;
7. Retention of mercury by soil components;
8. Formation of organomercurials.

Since publication of the above report, notes have been taken on any further publications related to mercury migration that may have appeared. It appears that there is a wealth of data on mercury concentrations in water, food and organic organisms, but only qualitative information on pathways and not very much on sorption mechanisms, particularly for elemental mercury (8-11).

LABORATORY WORK

As mentioned in the introduction, laboratory investigations have been directed towards several aspects of mercury migration. These are:

1. Solubility and/or entrainment of mercury in trench water and other fluids;
2. Mechanism of attachment to suspended solids and colloids and subsequent migration;
3. Mobility of elemental mercury in soil; and
4. Adsorption of trace mercury on structural materials.

Many of the uptake and adsorption tests involve relative concentrations, often in complex media, such as suspensions, which are most easily measured by radioactive tracers. On the other hand, to obtain direct measurements of very low concentrations in the parts-per-billion range, sensitive analytical facilities had to be available and for this purpose a cold-vapor cell atomic adsorption system was constructed, installed and calibrated. The latter work required considerable effort and will be discussed first.

Atomic Absorption Analysis of Mercury

A cold-vapor apparatus was designed which could be attached to an atomic adsorption spectrometer for detection of elemental mercury. The method is based on a procedure described by Hatch and Ott and improved by Hawley and Ingle (12). This method was chosen because it allows an inexpensive conversion of an existing atomic adsorption spectrometer into a mercury cold-vapor system, which is very reliable in the part per billion range. The short sample time and small sample size are useful in the routine work being done.

The results of this technique will be compared with the results obtained with the neutron activation analysis currently being prepared to relate the effectiveness of both techniques in their ability to detect mercury in the various experiments being carried out.

Experimental

Solution and Glassware Preparation

All solutions were prepared by using Fischer reagent grade chemicals and deionized water. Prior to making the standard solutions, the glassware to be used was soaked in a nitric acid - potassium permanganate solution

for 24 hours. Afterwards, the glassware was washed with strong glass cleansing detergent and rinsed 3 times with distilled water, HCL solution, HNO_3 solution, and again with distilled water, then dried in a drying oven for 2hr at 200°C. The main reason behind the extensive glassware preparation is that mercury in very dilute concentrations tends to adsorb onto the walls of the glass containers causing unwanted mercury loss. The nitric acid-potassium permanganate solution was one of many studied by Feldman (13) and suggested as being appropriate for protection against the mercury loss in the time period needed to run our calibrations. This solution keeps all mercury as Hg(II) ions and does not allow the mercury to be reduced to the Hg(O) state which absorbs onto the glassware.

A preservative containing 1%(^{v}/v)HNO_3 conc. and 0.02% (^{w}/v) $KMnO_4$ was used in preparing the standards which were made by serial dilutions of a Fischer stock mercury solution containing 1000 ppm. The standards were used immediately after preparation and were made fresh daily. The reductant was a $SnCl_2$ solution. All solution preparations are listed in Table I.

Instrumentation

The cold vapor atomic absorption procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. The carrier gas (air) flows

from a compressed air tank with a filter, attached to remove water condensation and particles in the air, through a 3-way stopcock, the reducing vessel, a drying tube, into the absorption cell, and is vented out the fume hood.

After the absorbance is recorded the 3-way stopcock valve is switched to the vacuum pump and the sample is flushed into the waste flask. The reducing vessel is then washed with deionized water and the valve is switched back to the air pump and another sample is ready.

The apparatus used is shown in Figure 1 with the instrument settings listed in Table 2.

The reduction vessel was constructed from a 11.5cm glass tube with a 1cm medium porous frit. There is a glass exit tube 2cm beneath the top, and 2 pointed impressions on the walls of the vessel to stop any bubbles from getting into the line.

The drying tube is made from a glass adapter with glass wool filled with $Mg(ClO_4)_2$ to keep moisture from entering the absorption cell.

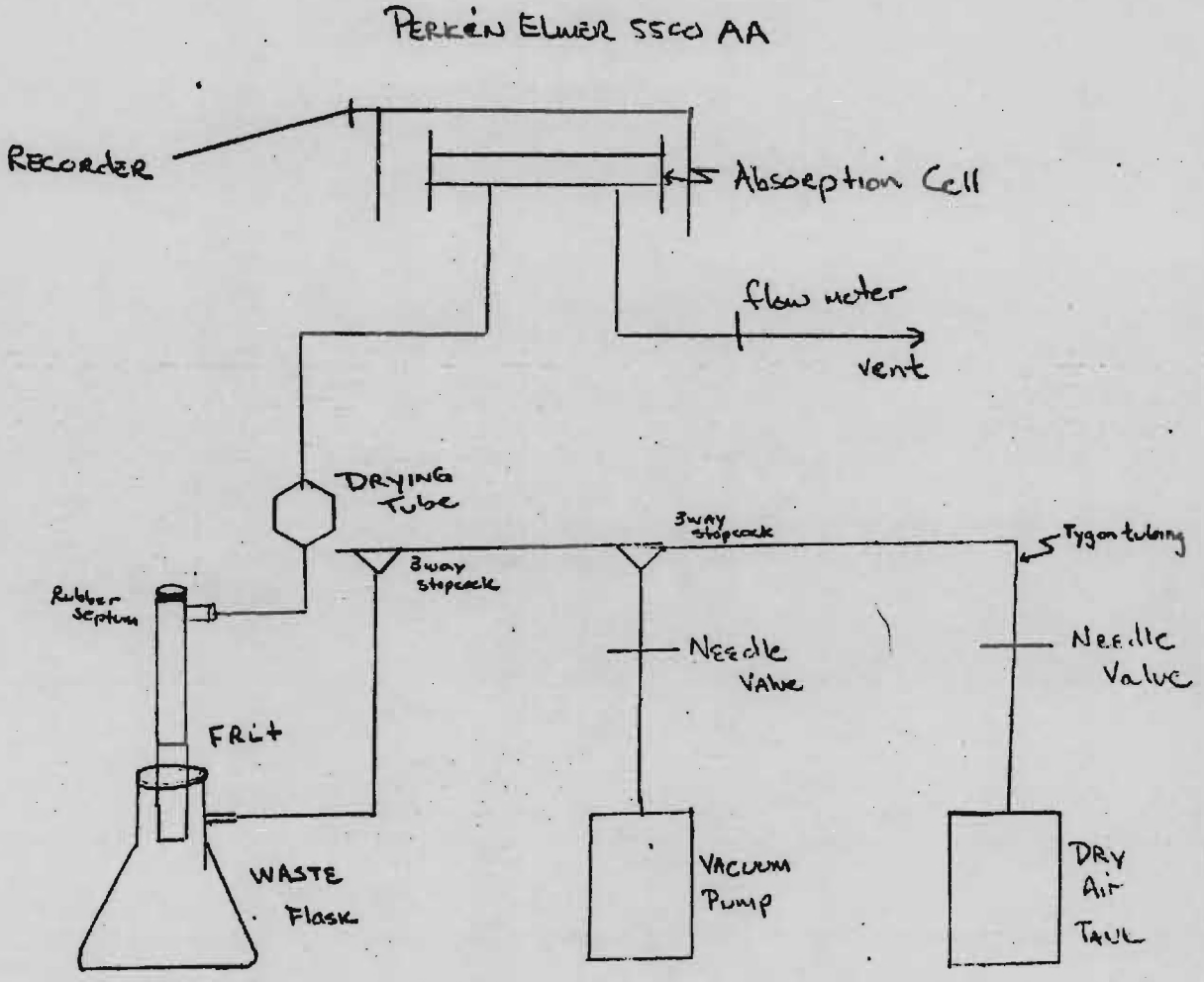


Figure 1 Mercury Cold Vapor Apparatus

Table I Solution Preparation

Reductant: 1g $SnCl_2$ + 1ml HCI (conc.) diluted to 100 ml 1% $^{w}/v$ $SnCl_2$

Oxidant: 0.2g $KMnO_4$ diluted to 100 ml [0.2% $^{w}/v$ $KMnO_4$]

Table 2 Optimal Values for Analysis

Flow rate	500 ml/min
Frit grade	Medium
Drying tube	4cm long x 10mm diameter $Mg(ClO_4)_2$
Gas carrier	Air
Volume of reductant	0.1 ml
Volume of sample	1.0 ml
Absorption cell	11.5cm
Radiation source	Hg hollow cathode lamp
Lamp current	8 mA
Slit width	H 0.7
Time constant	30 sec
Wavelength	253.7 nm

The drying tube connects into the tygon tubing and is constructed to allow for easy exchange because of the relatively high moisture content in the line. The tube is replaced after approximately 30 samples. The size of the tube is kept small to allow the mercury plug to remain compact to give a sharper absorbance peak and this also allows the flow rate to be kept at a desirable constant rate.

The absorption cell was constructed by attaching two quartz lenses to the end of the glass tubing. Inlet and outlet ports 3/16 inch o.d. were attached 2 cm from each end. For the alignment of the absorption cell it is mounted on the burner head of the Atomic Absorption Spectrometer using heavy duty duct tape. The absorption cell can then be properly aligned by adjusting the horizontal and vertical control knobs of the burner head. A flow meter is attached to the tygon tubing as it exits to the fume hood to allow for the adjustment of flow between sample runs to maintain a constant flow rate.

Operating Procedure

Instrumentation - The following outline is to be followed to prepare the Perkin Elmer 5500 for proper detection of mercury.

(1) Set P.E. 5500 to RUN

(2) Warm lamp, Press 8 [LAMP MA]

(3) Set wavelength, Press 253.7 [λ PEAK]

(4) Hit SETUP, You will get display of arbitrary NO. (45.55) Maximize Display: this done by adjusting the fine tuning knobs on top of the Hg cathode Lamp Hit SETUP again.

(5) Buttons Illuminated: AA ABS REC ABS PEAK HGHT

(6) Recorder: 1v FS: 12.5 cm/min ÷10 Push ON

(7) Set Zero by initiating Read Cycle
push READ
push AZ

(8) To Read, Push READ Record Pk. HGHT off the Display to Read Peak Area. Push [Peak AREA]; Push PK HGHT to get back into peak mode before running next sample.

Slits: H 0.7 Press 0.7 high

Time: 30s; Press 30 [t]

The optimal absorbance was found with the burner head control knobs at:
Vertical HGHT - 4.9
Horizontal - 5.0

Preparation of Mercury standards:

(1) Starting with 1000 mg/ml → 1000 ppm (A)

(2) Transfer using 1ml Eppendorf pipette, 5ml of (A) into 500ml volumetric flask: dilute to the mark with the HNO_3-$KMnO_4$ solution.
$\frac{5000\ \mu g}{500ml}$ = 10mg/ml → 10 ppm (B)

(3) Transfer 5ml of (B) into 500ml volumetric flask: dilute to the mark with the HNO_3-$KNnO_4$ solution
$\frac{50\ \mu g}{500ml} = \frac{0.1\ \mu g}{ml}$ = 100 ppb (C)

(4) Transfer 5ml of (C) into 50ml volumetric flask: dilute to the mark with the HNO_3-$KMnO_4$ solution
$\frac{0.5\mu g}{50ml}$ = 10ppb (D)

(5) Transfer 5ml of (C) into 25ml volumetric flask: dilute to the mark with the HNO_3-$KMnO_4$ solution
$\frac{0.5\mu g}{25ml}$ = 20ppb (E)

(6) Transfer 1ml of (B) into 50ml volumetric flask: dilute to the mark with the HNO_3-$KNnO_4$ solution
$\frac{10\mu g}{50ml}$ = 200 ppb (F)

(7) Transfer 0.5ml of (B) into 100ml volumetric flask: Dilute to the mark with the HNO_3-$KMnO_4$ solution
$\frac{5\mu g}{100ml}$ = 50ppb

Note: all dilutions are done using Eppendorf pipettes to ensure accuracy of measurement. Care must be used when measuring to avoid losing count of the number of milliliters which has already been transferred, since the largest Eppendorf can only transfer 1ml at a time. It is suggested to mark down on a piece of paper after each milliliter transferred. Also, practice using the Eppendorf pipettes prior to making the standard solutions.

PROCEDURE

The instrument is set up and the mercury standards prepared according to the procedure outlined earlier. The carrier gas is maintained at 500ml/min. 0.5 ml of the $SnCl_2$ reductant is injected into the reduction vessel with a Eppendorf pipette and the rubber septum is placed on top of the reduction vessel. This will reduce any Hg(0) present in the vessel prior to sample injection. After the recorder pen has returned to the baseline, and the flow rate is stable, 1ml of standard is injected into the septum with a sterilized disposable syringe.

When the Hg(II) standard is injected into the reduction vessel with the bubbling $SnCl_2$ solution, the Hg(II) ions are reduced to Hg(0) neutral atoms which diffuse from the solution into the carrier gas and are carried out of the reduction vessel through the drying tube into the absorption cell.

The peak absorbance is recorded on the recorder along with a direct readout of absorbance on the Perkin Elmer 5500. The total time for the pen to return back to baseline is approximately 20 sec. The sample is then evacuated by switching the 3 way stopcock to the vacuum pump position and a 1ml blank is flushed through the frit into the waste flask before the next sample is run.

A calibration curve is established by running each standard solution 3 times, starting with the least concentrated solution and working towards the highest concentration. This is done to avoid any memory effects which might occur from going from a large concentration to a smaller concentration. A new injection syringe is used for each concentration to avoid any contamination which might lead to a false reading.

A typical calibration curve is shown in Figure 2. The calibration is done before each set of samples to be run and checked again after the experiment to avoid any drifting in the system which might occur during the experiment and in between the time the next set of samples are run.

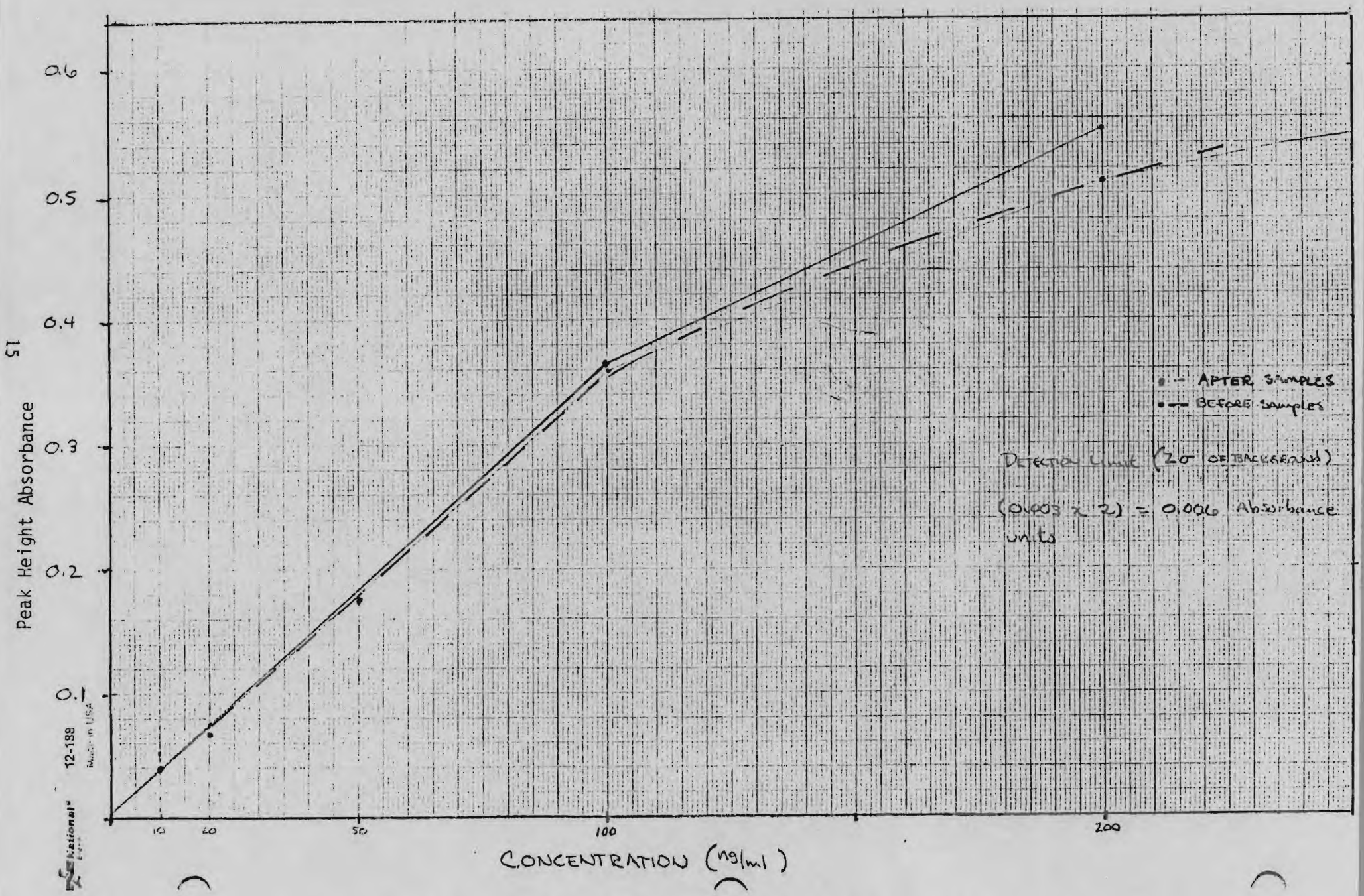


Figure 2 Calibration Curve - Run II (Peak height absorbance vs. Concentration)

Table 3. Calibration Data

Run I

Concentration (ng/ml)	Absorbance Peak HGHT	Peak Area
10	0.048	0.470
	0.041	0.430
	0.041	0.429
	0.043	0.421
50	0.174	1.284
	0.177	1.351
	0.177	1.377
	0.181	1.378
100	0.382	2.388
	0.349	2.547
	0.342	2.582
	0.345	2.519

Concentration	Peak HGHT	%error	Peak Area	%error
10	0.043±0.003	7.0	0.438±0.019	4.3
50	0.177±0.002	1.1	1.348±0.038	2.8
100	0.341±0.008	2.3	2.509±0.073	2.9

Background	Peak HGHT	Peak Area
Run I	0.006	0.182
	0.007	0.185
	0.007	0.187
	0.007	0.192
	0.007	0.201
	0.006	0.167
	0.0067±0.0005 (7.5%)	0.186±0.011 (6.1%)

Run II	Peak HGHT	Peak Area
	0.003	0.094
	0.003	0.099
	0.003	0.086
	0.003	0.083
	0.004	0.091
	0.003	0.089
	0.003	0.076
	0.003	0.076
	0.002	0.059
	0.003	0.074
	0.0031±0.0003 (10.7%)	0.083±0.012 (14.4%)

Calibration Data

Run II

Concentration (ng/ml)	Absorbance Peak HGHT	Peak Area
10	0.041	0.337
	0.041	0.347
	0.047	0.418
	0.047	0.408
	0.051	0.436
	0.048	0.406
20	0.067	0.549
	0.069	0.538
	0.069	0.528
	0.071	0.556
	0.066	0.526
100	0.349	3.008
	0.365	3.011
	0.362	3.157
200	0.506	3.746
	0.503	3.761
	0.503	3.572
	0.516	4.102
	0.529	4.230

Concentration	Peak HGHT	%error	Peak Area	%error
10	0.046±0.004	8.7	0.392±0.04	10.2
20	0.068±0.002	2.9	0.539±0.013	2.4
100	0.360±0.007	1.9	3.016±0.11	3.6
200	0.515±0.014	2.7	3.948±0.29	7.3

Calibration Data

Run III

Contamination Checks after Samples

Concentration	Peak HGHT	Peak Area
10	0.039	0.337
20	0.063	0.521
50	0.176	1.276±0.038 3%
100	0.342	2.610
200	0.555±0.0056	4.484

Calibration Results

The results of the calibration are shown in Figures 2 through 5. Figures 2 and 3 were the results from the first calibration runs, whereas Figures 4 and 5 were results from a second calibration. All the results obtained were quite linear up to the 100 ng/ml level with a relative standard deviation of 0.002 at the 50 ng/ml level.

The detection limit was found to be 0.005 and 0.006 PEAK HEIGHT absorbance units for Run 1 and Run 2 respectively. This corresponds to approximately 0.05 - 1 ppb, which is far below the range of interest for the experiments being done.

Both the peak height and peak area can be obtained from the Perkin Elmer 5500. The peak area is a more precise determination of absorbance since it measures the number of atoms under the curve and is not dependent on the flow rate and method of injection. The peak height was recorded primarily as a means of showing the reproducibility of the peaks at each concentration. Figure 6 shows the peaks as they were recorded for each concentration during the first trial calibration.

The main source of imprecision with the system, which can be seen from the difference in fluctuation of the peak areas, especially up to 10 ng/ml, is the method of injection. The 1cc disposable syringe which was used to

inject the sample, allows adequate reproducibility of results for evaluating the design and reproducibility of the system. The consistency or precision of the apparatus could probably be further increased by using a more accurate syringe, similar to those used in gas chromatography. The benefit of using disposable syringes is that they are inexpensive and can be thrown away after each injection at a particular concentration to avoid contamination. The more accurate G.C. syringes could be rinsed with the oxidizing solution ($KMNO_4$) and reducing agents $SnCl_2$ after each set of samples to avoid contamination, and then a blank (H_2O) sample could be run to assure that no mercury residue was left in the syringe.

In conclusion, the cold vapor apparatus is quite adequate for the type of samples being prepared by our group, and further work is being done to allow for a higher sensitivity and precision to be achieved. (14,15).

Samples

A series of samples is currently being tested to observe the difference in solubility of mercury in various solutions as a function of time and temperature.

Four different solutions are prepared using highly distilled mercury, Fischer grade Kerosene, high purity deionized water in 30 nl serum vials which are capped with a rubber septum and sealed with an aluminum cap to avoid contamination.

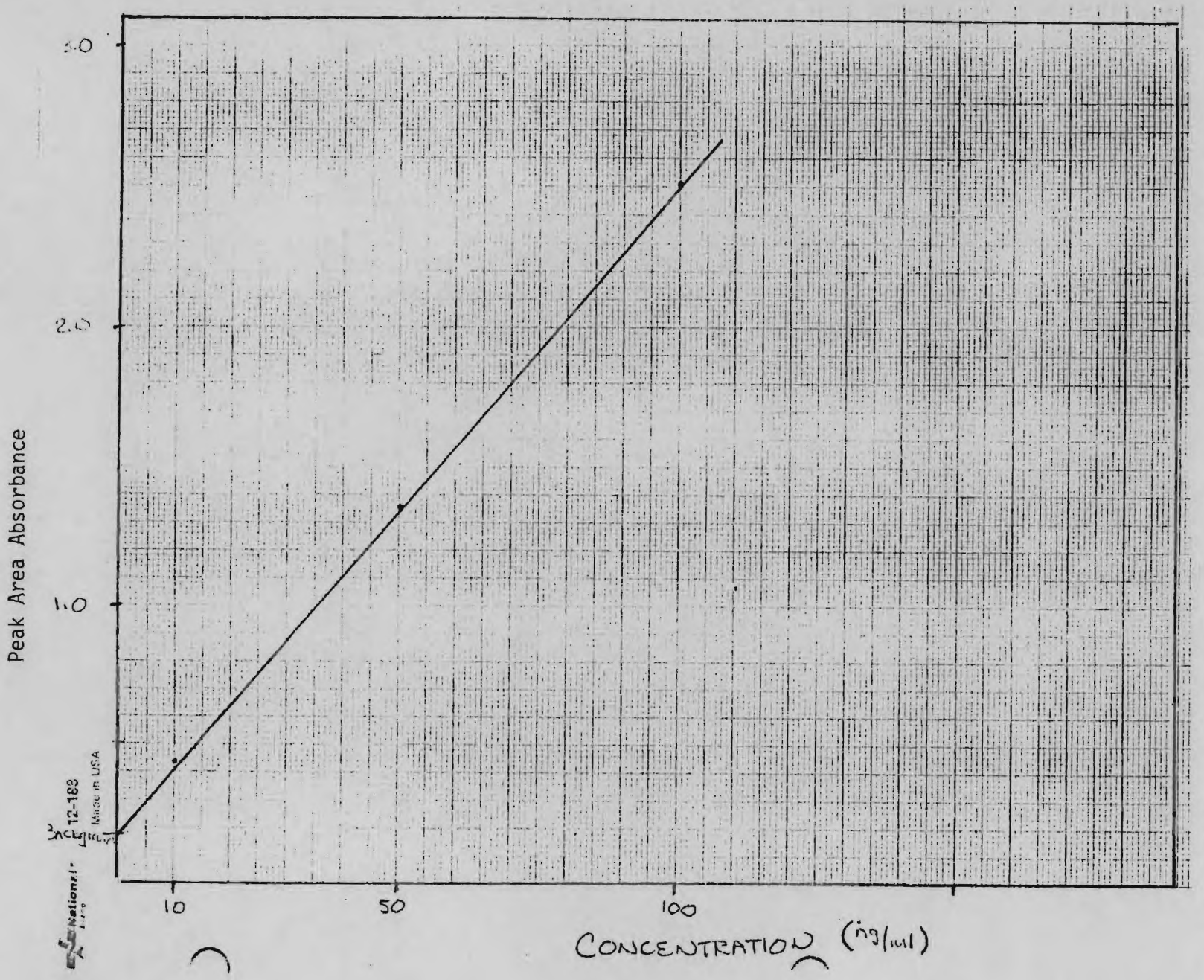


Figure 3 Calibration Curve - Run I (Peak area absorbance vs.Concentration)

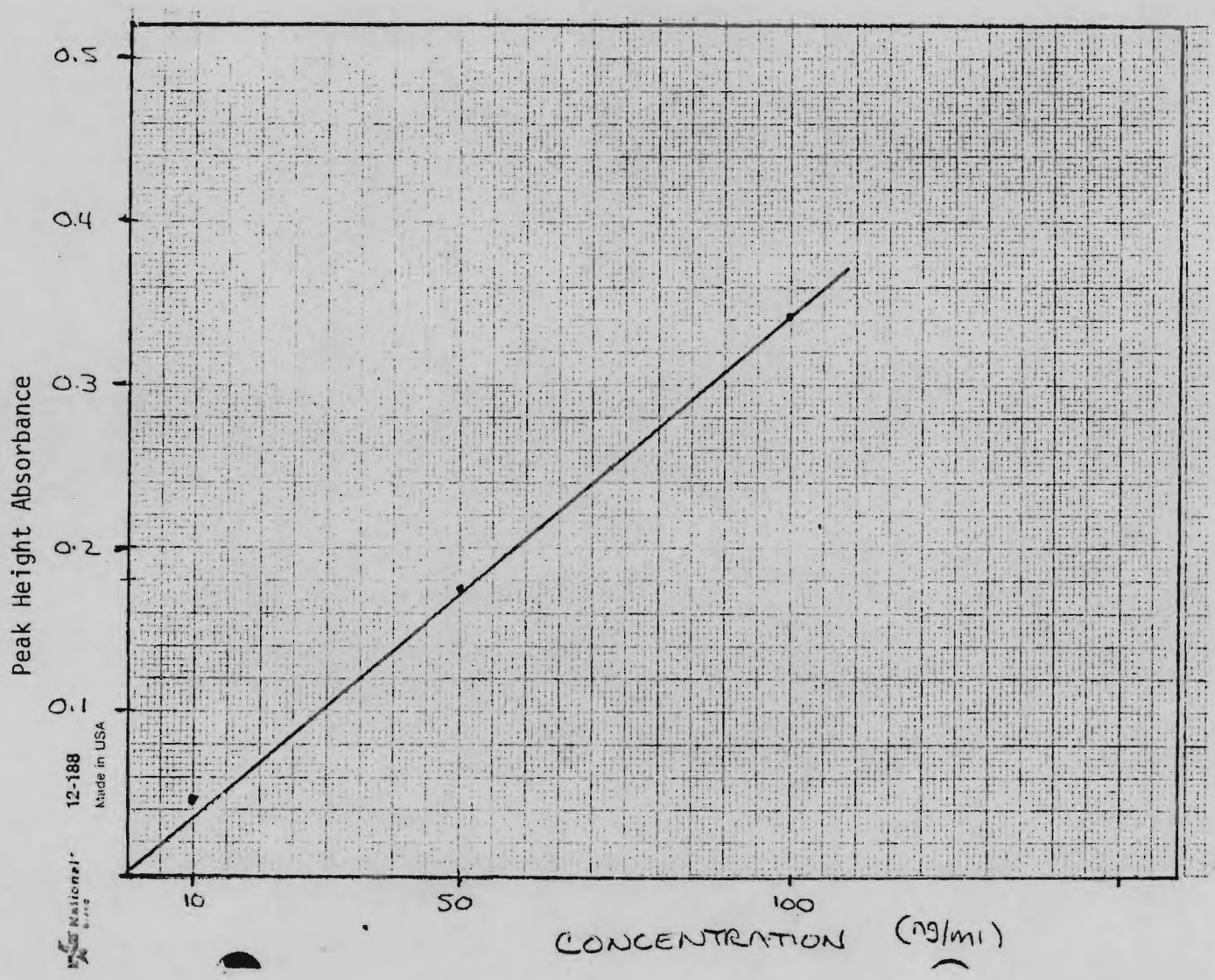


Figure 4 Calibration Curve - Run I

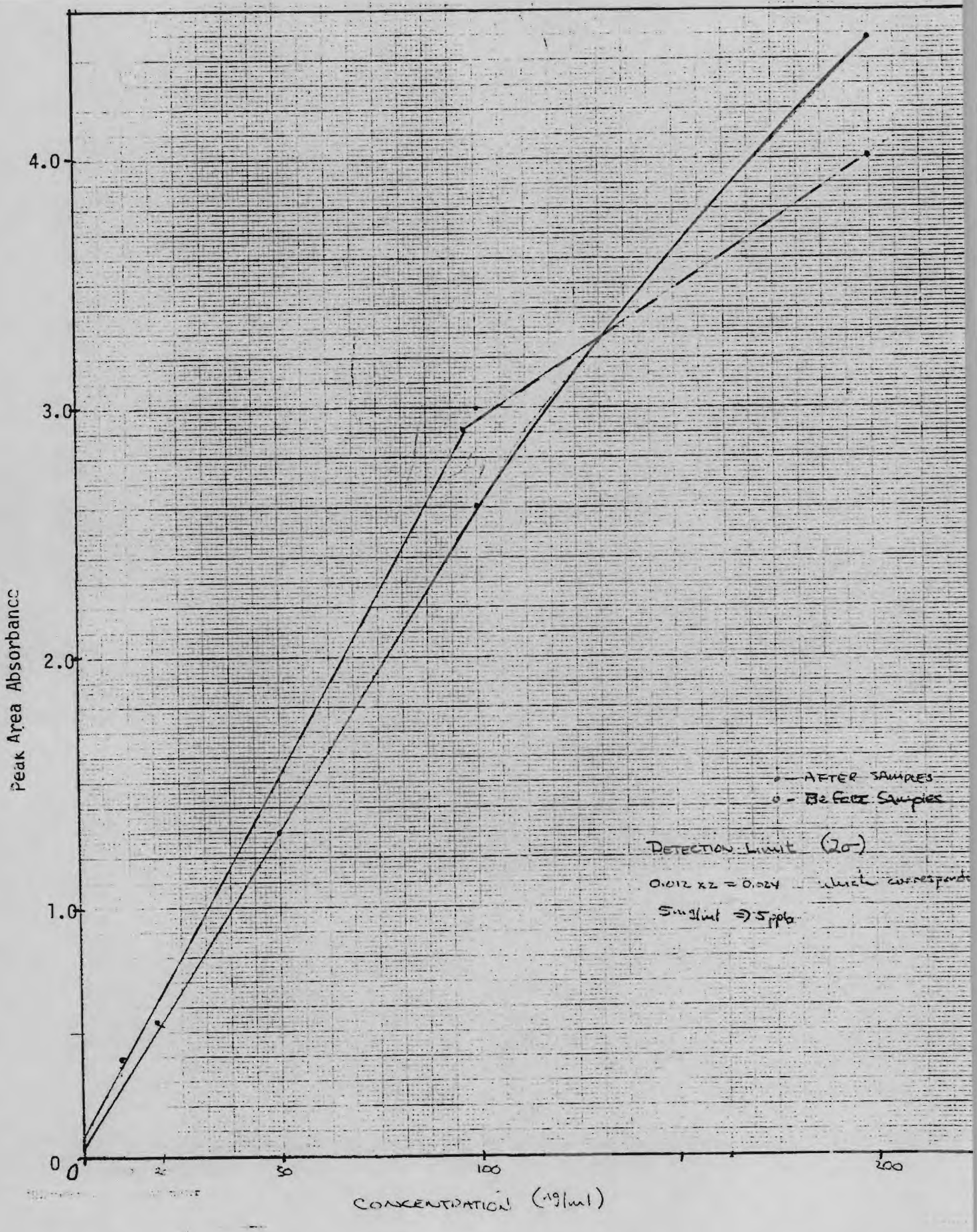


Figure 5 Calibration Curve - Run II
(Peak Area Absorbance vs. Concentration)

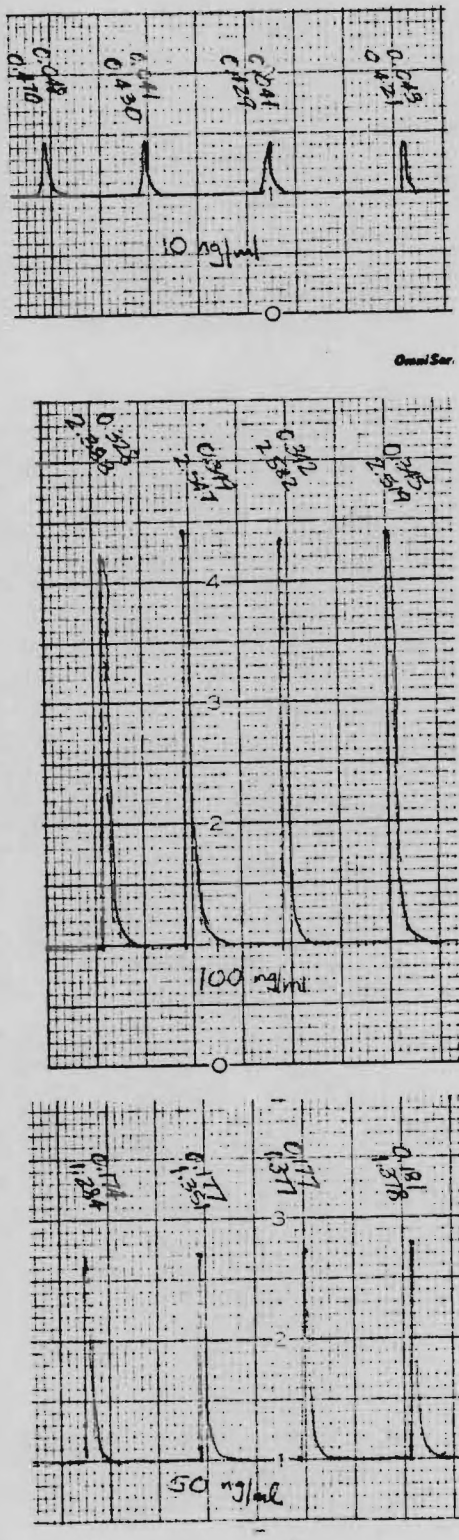
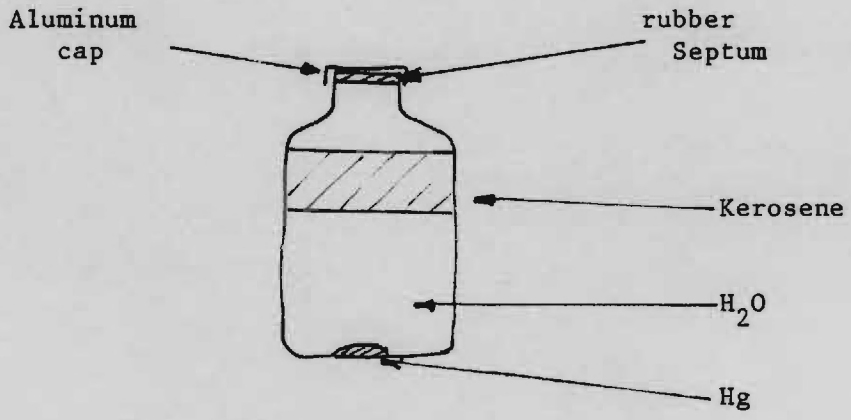


Figure 6 Reproducibility of Standards

SAMPLE	CONTENTS
A	Hg(O) 1g + H_2O 15ml + Kerosene 3ml
B	Hg(O) 1g + H_2O 15ml
C	H_2O 15ml + Kerosene 3ml
D	H_2O 15ml

A trial run was done on these samples and the results are enclosed but the amount of Hg was not adequately measured so the results are meaningless.



Example of Serum Vial Setup

Data for Mercury Solubility Trial Run

SAMPLE	PEAK HEIGHT
A	0.687
B	0.432
C	0.017
D	0.015

Radioactive Tracers

Since the measurement of partition factors is most easily done with radioactive tracers, quantities of triple-distilled mercury were prepared and flamed sealed in quartz tubes. Two samples of 1cc (13.5gm) and one sample of 0.1 cc were prepared. The smaller sample was irradiated first, to verify the absence of significant amounts of impurity and associated radiation levels post exposure in the reactor.

After irradiation the small sample had a radiation level for the glass and sample of $\sim$ 150 mR/hr. Based on this reading the larger sample was irradiated for 15 minutes at 10^{13} n/sec-cm^2 (as was the first sample). Immediately after removal from the reactor the radiation level for the sample, glass, and plastic container was $\sim$ 500 mR/hr. This level was in agreement with calculations done prior. The expected exposure rate from just the mercury was 250 mR/hr.

Three days later the sample was assayed with a germanium detector for the presence of unexpected isotopes. No peaks occured over the energy range of 0 to 1.5 MeV other than Hg^{19}, Hg^{197m}, and Hg^{203}. As expected, Hg^{203} was the dominant peak due to its long half-life relative to other Hg isotopes.

A third sample was not irradiated and has been saved for future uses. Both the third sample and the activated sample have been stored in the lead shield in the corner of lab 109. The Hg is contained in a plastic vial inside a small orange lead pig. The "cold" sample is also in the shield in an open plastic container.

Seepage Tests

The simplest and most primitive notion of migration involves seepage of small mercury droplets through coarse soil material. Orebaugh and Hale (6) showed that in most cases such movement probably would entail volatile mercury vapor diffusion with subsequent condensation, a slow and inefficient process at subsurface temperatures. Turner (16) has done some simple calculations to relate pore entry radii with mercury pressure. For pore radii below 10 microns entry will require pressures in excess of 20 cm of mercury (column height); this assumes surface tension values of nominally pure mercury.

For experimental verification a number of packed columns were prepared in 3cm diam. glass tubes, using different sand and soil samples that had been characterized for the Lysimeter project. The surfaces of the columns were carefully leveled and smoothed. About 1 mm of clean mercury was carefully deposited and left standing. After several months standing, it is proposed to freeze the columns and disect them to measure the degree of mercury penetration if any.

Table 5 lists the condition of the various columns. "Wet" soil was prepared by placing 50g of sand or soil in a beaker, covering it with water, and draining the excess. After long periods of standing it is assumed that each wet column contains only the appropriate minimum residual moisture concentration.

TABLE 5. Details on Seepage Columns

TYPE	SIZE	CONDITION	AMOUNT OF MERCURY ON TOP OF SOIL
SRP #1	20	DRY	41.829 g
SRP #1	40	DRY	41.774 g
SRP #1	60	DRY	42.210 g
SRP #2	20	DRY	43.109 g
SRP #2	40	DRY	44.386 g
SRP #2	60	DRY	44.710 g
SAND	20	DRY	44.044 g
SAND	40	DRY	43.668 g
SAND	60	DRY	44.050 g
SRP #1	20	WET	43.983 g
SRP #1	40	WET	43.243 g
SRP #1	60	WET	42.968 g
SRP #2	20	WET	43.861 g
SRP #2	40	WET	41.356 g
SRP #2	60	WET	43.682 g
SAND	20	WET	40.282 g
SAND	40	WET	41.959 g
SAND	60	WET	42.321 g

MIXING AND SOLUBILITY TESTS

Only qualitative tests have been done in this area; it is expected to obtain more positive results early in 1985, now that analytical procedures are in place.

A first set of experiments was performed to see if mercury and kaolin in aqueous suspension would mix. A magnetic stirrer, with 52.715 g mercury in 50 ml of kaolin solution, and a bubbler, with 135.259 g of mercury in 500 ml of kaolin solution, were set up. After approximately 20.5 hours, these devices were shut off and it was noted that while both solutes were grayish (denoting mercury in both), the magnetic stirrer solute was darker than the bubbler solute. After settling for one day, the solutes were removed and examined microscopically for mercury. The experimenter was not able to distinguish between the plain kaolin solution and the solute samples. With a fresh batch of kaolin solution in the bubbler, samples were taken every 15 minutes (from 0 to 255 minutes) to try and detect mercury dispersion into the solution; once again, none was noted visually. This procedure was repeated and the samples then taken were saved for subsequent AA analysis. However, the nature of the sample indicated the analytical problems that must be anticipated for such heterogeneous samples. In the long run, this type of test is probably done more readily with radiotracers. It should also be noted that the stirring of the above samples was much more vigorous than would occur in nature.

Another subject being investigated is the solubility of mercury in various liquids. The basic method of these test was to take a small amount of mercury (about 6 ml) and add it to a larger amount of the liquid of interest. This mix is then agitated slowly for a number of days to allow it to reach saturation. Samples were then taken at time t = 0,5,10,30,60 minutes after agitation was stopped. This was done to note any differences in concentration between the first and the subsequent samples; such difference which could then be ascribed to the effect of gravity on the suspended particles, not on a true solution.

Liquids used in this determination were:

1) Kerosene	(Samples A1-A5)
2) Distilled Water	(Samples B1-B5)
3) EDTA-Distilled Water Solution	(Samples C1-C5)
4) Sand Equilibrated Water	(Samples D1-D5)
5) SRP Soil #1 Equilibrated Water	(Samples E1-E5)
6) SRP Soil #2 Equilibrated Water	(Samples F1-F5)

Preliminary AA analysis indicated that the mercury levels were too high for the system, which was completely overloaded. Future tests will have to employ small amounts of mercury. Since kerosene is not a satisfactory medium for AA analysis, kerosene uptake will have to be deduced from radiotracer tests.

ADSORPTION ON STRUCTURAL MATERIALS

During the summer of 1984 the need arose to select materials that would be suitable for wall material in lysimeters that were proposed for possible future investigations. Tests were initiated, therefore, to check the adsorption or surface attachment of mercury to various candidate materials. These tests are continuing and only preliminary results are at hand.

The initial experimental set-up used in this investigation consisted of a coupon of various materials held by a black rubber stopper on the end of a glass rod being slowly rotated in a beaker containing elemental mercury. The initial tests with this set-up were performed with Teflon, polyethylene, glass, and galvanized steel coupons. Visual inspection of these samples after 29 days in the mercury showed a heavy deposition of mercury on the galvanized steel, a small amount on the polyethylene and Teflon, and an amount intermediate between these on the glass coupon.

Several problems were encountered with this technique. Upon disassembly of this set-up and preparation for subsequent tests, it was noticed that there was a dull, blackish, dirty deposit layer on the top of the mercury. This was realized to be a problem, as this film would cling to the coupon as it is removed from the solution and yield a variable and artificially high determination of elemental mercury adsorption to these materials.

Subsequent investigations, in which two beakers containing clean elemental mercury were prepared and a black rubber was placed into one and rotated (as in the initial experimental set-up), revealed that the stopper was partially responsible for this dirty deposit, as after 6 days, the beaker with the stopper showed a black deposit similar to that seen previously, and the other beaker showed only a small amount of a dull oxide layer. As a result of this determination, it was decided to make the coupon holder out of polyethylene, which caused no such effect.

Another problem which was encountered was in the prevention of the formation of the oxide layer on the top of the mercury exposed to air. Several beakers containing filtered clean mercury were set up and a layer of various liquids was poured on top of the mercury to isolate it from air to prevent the formation of this oxide. The best success was obtained with a pure mineral oil, as the top of the sample covered by a layer of this remained shiny and clear for the 2 weeks that these samples were observed. Also, there was no evaporation problem with the oil as was observed with some of the other liquids after a few days.

The oil layer that will form on the coupon as it is withdrawn from the sample beaker covered with an oil layer will not be a problem with the quantitative determination of mercury adsorption using activated mercury and a counting technique, but it may be a problem with the cold vapor technique. Work needs to be done to determine the effect of this interference if this oil layer is used with samples to be analyzed using the cold-vapor technique.

An experimental set-up was constructed that made it possible to use four separate coupons in the same mercury sample. The coupon holders are attached to the underside of a circular piece of plastic, which itself is attached to a glass rod to permit slow rotation of this assembly in the mercury sample. The coupon holders were fashioned out of polyethylene tubing connectors, with a slit cut in the polyethylene to enable it to hold the coupon.

An activated elemental mercury solution was prepared from the 1mCi (originally) of mercury activated on 8/17/84 in the Georgia Tech Research Reactor in approximately 500ml of filtered clean mercury on November 2, 1984. With this activated sample, two sets of adsorption tests were performed. The first set consisted of two fiberglass coupons (approximately 13/8" x 1" x 1/32") and 2 galvanized steel coupons (approximately 1 3/8" x 1" x 1/16"). The second set, used 2 aluminum coupons (approximately 1 1/4" x 1" x 1/32) and one each of the fiberglass and galvanized coupons. Each of these sets remained in the activated solution for 6 days and the apparatus was slowly rotated in the solution at about 5 rpm in each test.

The samples were counted on December 1, 1984, which was done by integrating under the 0.279 Mev photopeak of Hg-203 (all other short-lived mercury activation products have decayed to negligible levels). The results are summarized in Table 6.

Table 6 - Count Results of Hg Adsorption Tests

Detector: Canberra GeLi Detector, Serial No. 941R. Operating Voltage=2000V
MCA: Canberra Omega I GIT #0051255
Amplifier: ORTEC Model 485 GIT #0069180
Power Supply: Canberra Model 3005 GIT #0048970

Galvanized Steel	Gross Count	Avg. Bkgd	Net Count
1	3361	109	2053
2	3080	115	1700
3	3412	116	2020

Peak Channels 90-102
Count time = 200 sec.

Fiberglass	Gross Count	Avg. Bkgd	Net Count
1	2074	98	898
2	2183	104	935
3	2007	102	783

Peak Channels 90-102
Count time = 200 sec.

Aluminum	Gross Count	Avg. Bkgd.	Net Count
1	2691	105	1431
2	2412	107	1128

Peak Channels 90-102
Count time = 200 sec.

CONCLUSIONS

As mentioned in the Introduction, this report is a progress report on ongoing work. Only the literature review is considered concluded. Work in the other areas has involved the setting up of the cold-vapor atomic absorption system, which is now working and has been calibrated. Other activities are in hand and it is expected that useful results can be reported at an early date.

Close contact has been maintained with technical staff at the Savannah River Laboratory and we wish to acknowledge the helpful coopeation of Dr. John A. Stone and Dr. Steven B. Oblath of the Waste Technology Division of SRL.

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